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PART D

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DUAL CAPILLARY COLUMN ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBs) IN DRINKING WATERS.

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Increased public awareness of the affects of chronic exposure to PCBs has sparked the need for fast accurate methods for quantitating PCBs in environmental samples. Original Ministry of the Environment quantitation methods involved solvent extraction followed by either perchlorination of all biphenyl to decachlorobiphenyl and its subsequent analysis or the quantitation and grouping of PCB peaks generated by single packed column Gas Chromatographic (GC) analysis. The advent of capillary columns allowed for much greater resolution of individual PCB isomers and more accurate quantitation. Further enhancement of PCB quantitation techniques has been accomplished by using dual fused silica capillary column methodology for quantitation and confirmation of results. Dual capillary column PCB quantitation methodology for the analysis of drinking water and drinking water sources was developed using PCB standards and fortified water samples.

Wet chemistry preparation of samples for GC analysis was similar to that employed for organochlorine pesticides and consisted of liquid/liquid extraction, concentration by rotovapor apparatus, wet pack florisil cleanup and final concentration by vortex evaporation. Sample extracts containing organic chlorine pesticides such as DDT and DDD along with PCBs were resplit using dry pack florisil cleanup and reanalysed.

Analytical methods were developed using a Varian 6000 Gas Chromatograph, equipped with a split/splitless injector and dual Electron Capture Detectors (ECD), coupled with a Varian Vista 402 data station. Sample extracts, split on injection, travel through two 30m 0.25 mm ID fused silica capillary columns of differring polarity (DB1/DB1701) before passing through the ECD detectors.

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Two dual capillary column methods differing only in instrument calibration mixture were tested. Calibration mixtures containing Aroclors 1254 and 1260 were chosen because >95% of PCB positive samples received by the Drinking Water Section Organic Water Unit contain these Aroclors.

Method PCBMIX, using A 1:1 mixture of Aroclors 1254 and 1260 for calibration, was determined to be better than method PCB using Aroclor 1254 only. Method PCBMIX was much better for quantitating mixed PCB extracts although neither method was adequate when quantitating extracts containing large proportions of Aroclors other than 1254 and 1260. Instrument reproducibility using method PCBMIX was 3% with a linear range of 20 to 500 ng/ml. Spike recoveries averaged 107% ± 8% over the 20 ng/l to 2000 ng/l range tested.

Method PCBMIX was checked and validated against current Ministry of the Environment single packed column PCB quantitation methodology using laboratory standards and fortified water samples as well as real environmental drinking water samples. Results obtained were comparable. No significant difference was found between the quantitation methods although method PCBMIX results tended to be on average slightly higher.